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TEM investigation of the microstructure in a single crystal nickelbase superalloy creep tested in [011] orientation

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ABSTRACT

Quantitative TEM investigations are performed on [011] oriented single crystals of Nickelbase superalloys creep tested at 1033 K under a load of 680 MPa. Multiple slip in the matrix occurs on four $1/2\langle 110 \rangle \{111\}$ slip systems from beginning on. Octahedral cross slip is not activated. Cube slip does not occur, whereas during secondary creep screw interfacial dislocation cross slip in the cube γ/γ' -interface. In the matrix phase, deformation is concentrated in the "roof" channels. Calculations show that this is the result of the superposition of coherency and external stresses in small matrix channels. In the stage of secondary creep, γ' precipitates are sheared by Shockley superpartials. Common shearing of matrix and γ' particles by Shockley superpartials has been analysed. Continued common shear on adjacent $\{111\}$ planes finally causes damage by mechanical twinning. The high creep rate in the [011] orientation is considered to be caused by the stress concentration in roof matrix channels and the few activated slip systems.

1. INTRODUCTION

The strong creep anisotropy of single crystal Nickelbase superalloys at intermediate temperatures has been investigated by several authors [1,2,3,4]. The results were discussed considering the lattice rotations needed to activate multiple slip [2] or in terms of cubic slip [5].

In order to document the evolution of the microstructure during creep, in this paper, intense quantitative TEM investigations were performed on SRR99 in the early stages of creep. The experimental results are compared with calculations of stress distributions in the microstructure.

2. EXPERIMENTAL

At 1033K and a constant load of 680MPa, tensile creep tests were performed on two single crystals of SRR99 in [011] orientation. The crystals had received a heat treatment consisting of solutioning (1h at 1553K + 2h at 1563K + 0.5h at 1573K + 0.5h at 1578K), cooling down to 1273K with a cooling rate of 0.7Ks^{-1} and subsequent air cooling followed by precipitation annealing (4h at 1353K + 16h at 1143K). After this heat treatment, the alloy contains a volume fraction of 70% of the ordered γ' phase. The mean length of the γ' cubes is 450nm, the width of the matrix channels is about 50nm. The cubic morphology of the γ' phase was maintained throughout the tests.

One of the creep tests was interrupted after 20h with a plastic elongation of $\epsilon_p = 0.46\%$. The second creep test was interrupted just beyond the minimum of the creep rate at a plastic strain of $\epsilon_p = 0.075\%$ which was reached after 0.75 h. The dependence of the creep rate $\dot{\epsilon}$ from plastic strain ϵ_p is depicted in Fig. 1.

Additionally, the microstructure of a ruptured [011] single crystal alloy CMSX-6 ($\epsilon_p = 9.7\%$, $\sigma_L = 680\text{MPa}$) was analysed. The investigations were done using a Philips CM-30 TEM operated at 300kV.

3. RESULTS

3.1 Dislocation structure after $\epsilon_p = 0.075\%$

An overview of the microstructure is given in Fig. 2 showing interfacial dislocations in matrix channels oriented under 45° to the load axis. These channels will be denoted as roof channels in contrast to the vertical channels that contain the load axis. The dislocation density in roof channels is considerably higher than in vertical channels. In the roof channels slip proceeds on the four octahedral system having the highest Schmid factors. The slip planes are (111) and ($\bar{1}\bar{1}\bar{1}$). Octahedral cross slip is not activated. During slip of matrix dislocations dipoles with $\langle 110 \rangle$ interfacial line segments are created. Due to the absence of any cross slip and climb at this stage of creep, the dipole habit planes are identical with the $\{111\}$ slip planes. Shearing of the γ' phase is a very rare event. Slip on cubic planes was not observed.

3.2 Dislocation structure after $\epsilon_p = 0.46\%$

In the secondary stage of creep, shear in matrix and the γ' phase by super stacking faults (SSF) is observed on the same slip planes (Fig. 3). Determination of the dipole habit planes revealed that after 20 hours of creep interfacial screw segments had cross slipped in the cube γ/γ' interfaces, whereas mixed dislocations do not climb and therefore still lie on their slip planes.

Analysis of the SSF showed that the dissociation of matrix dislocations occurs by the single dislocation mechanism [6] as well as by the two dislocation process [7]. Cross slip of dipole segments in the γ/γ' interfaces was observed to ease the two dislocation mechanism. In both cases shear of the γ' particle is completed by the recombination of super Shockley and Shockley partial to a matrix dislocation at the exit side of the precipitate. In addition to these well known configurations common shear of γ' phase and matrix by the same $1/3\langle 112 \rangle$ super Shockley partial was analysed.

Fig. 4 shows a super stacking fault that has leaped from one γ' cube over the matrix into the second precipitate. The analysed configuration is depicted schematically in Fig. 5b.

3.3 Dislocation structure after rupture

Far from the fracture area, the microstructure is similar to that in the secondary stage of creep. Near the fracture area however, numerous mechanical twins running through matrix and γ' phase were observed to be the final damage mechanism. Fig. 6 shows that at the end of creep in this orientation plastic deformation is obtained by twinning on the $(\bar{1}11)$ plane. We consider this as a result of continued common shear on adjacent $\{111\}$ planes. The restriction to only one slip plane at the end of deformation is assisted by the tendency to planar deformation due to few slip systems.

3.4 Calculation of resolved shear stresses in the matrix

In order to account for the observed concentration of matrix slip in the roof channels, resolved shear stresses in differently oriented channels have been calculated. Due to the superposition of external load σ_L and coherency stresses σ_c , the resolved shear stresses available to expand a dipole with 60° interfacial segments are $1/\sqrt{6}(\sigma_{cn} - \sigma_{cp} + \sigma_L) = 360\text{MPa}$ in the roof matrix channels and $1/\sqrt{6}(-\sigma_{cn} + \sigma_{cp} + \sigma_L) = 230\text{MPa}$ in vertical matrix channels. Dipoles with screw interfacial dislocations in roof channels are not effected by coherency stresses and therefore the resolved shear stress is $1/\sqrt{6} \cdot \sigma_L = 280\text{MPa}$. Screw interfacial dislocations in vertical channels experience no shear stress. The values of the coherency stress components σ_{cn} and σ_{cp} normal and parallel to the γ/γ' interface were taken from calculations by FEM [8].

The critical resolved shear stress needed for the formation of matrix dipoles as a function of the matrix channel width was calculated using a computer model based on anisotropic elastic theory [9]. The results are shown in Fig. 7. It is seen that for screw and 60° interfacial dislocations in roof channels the critical matrix channel width is smaller than the mean channel width of 50nm: The shear stress is high enough for dipole expansion in matrix channels. In contrast, the resolved shear stress acting on dipoles with 60° interfacial segments in vertical channels is insufficient to expand these dipoles in matrix channels of 50 nm width. These results explain the differences between the dislocation densities in roof and vertical channels. The concentration of internal stresses in the roof channels is considered to be the main reason for the high creep rate in the $[011]$ orientation.

4. SUMMARY

- Deformation starts in the matrix on four slip systems activated on (111) and $(\bar{1}11)$ planes.
- In the $[011]$ orientation $\{111\}$ cross slip is not activated.
- Due to the superposition of external and coherency stresses, different resolved shear stresses act on dipoles with screw and mixed interfacial dislocations in roof channels as compared to vertical channels. Therefore slip is concentrated in matrix roof channels.
- At later stages of creep, screw interfacial dislocations cross slip in cubic γ/γ' interfaces.
- Secondary creep is characterised by the additional shearing of the γ' phase by Shockley super partials on the same slip planes.
- In addition to the dissociation of matrix dislocations at γ/γ' interfaces and their subsequent recombination at the exit surface of the precipitate, common shearing of γ' phase and matrix by Shockley super partials was analysed.
- Final damage is caused by mechanical twinning of matrix and γ' phase.
- No cubic slip is observed in matrix or γ' volume.
- Our results show that the theories of single slip or cubic slip are inadequate to account for creep anisotropy.

- The high creep rate and the short rupture lives in the [011] orientation is related to the stress accumulation in matrix roof channels and the general tendency of planar shear due to the few activated slip systems.

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Fig. 1: Creep rate as function of strain
SRR99, $T = 1033\text{K}$, $\sigma_L = 680\text{MPa}$

Fig. 2: Overview of the microstructure close to the creep rate minimum. $\epsilon_p = 0.075\%$

Fig. 3: Overview of the microstructure in the stage of secondary creep $\varepsilon_p = 0.46\%$, $\vec{k}_0 = [0\bar{1}1]$, (111) and $(\bar{1}11)$ edge dislocation on.

Fig. 4: Common shear of two γ' cubes and matrix during secondary creep by $1/3\langle 112 \rangle$ dislocation. $\vec{k}_0 = [011]$, $\vec{g} = (1\bar{1}1)$

Fig. 5: Schematic sketch of the succession of common shear

Fig. 6: Mechanical twinning in ruptured specimen
 $\epsilon_p = 9.7\%$. $\vec{k}_0 = [0\bar{1}1]$, $\vec{g} = (111)_{\text{Twin}}$

Fig. 7: Critical shear stress for the expansion of a dipole loop in a matrix channel of width l .